Design and Fabrication of Vibrating Sample Magnetometer.

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Design and Fabrication of Vibrating Sample Magnetometer.

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CERIFICATE OF APPROVAL

This is to certify that the work in the thesis entitled **Design and Fabrication of Vibrating Sample Magnetometer** submitted by **Debi Prasad Pattnaik** is a record of original research work and has been carried out under my supervision and guidance in partial fulfillment of the requirements for the award of the Degree of **Integrated Masters of Science in Physics** at National Institute of Technology, Rourkela, and this work has not been submitted elsewhere before for any other academic degree or award

Rourkela

Date: 10^{th} May, 2014

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Dr. P. N Vishwakarma.

DECLARATION

This thesis entitled, "**Design and Fabrication of Vibrating Sample Magnetometer**" is a presentation of my original research work. Wherever contributions of others are involved, every effort is made to indicate this clearly, with due reference to the literature.

The work was done under the guidance of Dr. Prakash Nath Vishwakarma at National Institute of Technology Rourkela, India.

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Debi prasad Pattnaik .

Debi Prasad Pattnaik

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Abstract

A simple Vibrating Sample Magnetometer (VSM) has been designed, constructed and developed for the measurement of magnetization. The design was mainly was aimed at improving the sensitivity of the instrument so a large number of coil designs, from 2 coil cylinder system, 8 coil cube arrangement to 2 coil parallel system , relays were employed. The vibration unit comprised a normal stereo speaker of 4 ohms impedance which provided the necessary harmonic vibration. A laboratory electromagnet of field strength 0.25 Tesla is used to provide the magnetic field. The induced voltage signal is measured using a Lock In Amplifier (SR830) interfaced to the personal computer using Lab view. The temperature is monitored using Pt-100 temperature sensors by tabulating their resistance at different temperatures. The VSM is calibrated against a standard Nickel sample. The VSM has been used to study the magnetic properties of magnetic materials.

Chapter 1

Introduction

> "A wonder I experienced as a child of 4 or 5 years, when my father showed me a compass. That this needle behaved in such a determined way did not at all fit into the nature of events. I can still remember — or at least I believe I can remember — that this experience made a deep and lasting impression on me. " -Albert Einstein

Magnetism, is regarded as the oldest technolgy and a magical science. It was first recorded around 600 BC by the Greeks studying ferrite rocks (lodestone). The earliest mention of a magnetic compass used for navigation is from a Chinese text dated 1040–1044 A.D., but it may have been invented there much earlier. It was apparently first used for orientation on land, not at sea.An understanding of the relationship between electricity and magnetism began in 1819 with work by Hans Christian Oersted, who discovered more or less by accident that an electric current could influence a compass needle. This landmark experiment is known as Oersted's Experiment. Several other experiments followed to establish more firm relationships.Today it is one of the most important properties of nature and is used in a multitude of technological devices from power plants to computer chips. This is largely due to the interaction of magnets with electricity, which is used to make electric motors to electric drives to bio-medical equipments and an infinite array of sophisiticated and sensitive instruments and devices . This, however, has been the result of years worth of investment in the research and development of magnetic materials . Seen in the broader historical perspective, it is the intimate connection between discovery and practical use which causes most of the magic of magnetism, and it is certainly illustrative for the magic of science in general.

1.1 Origins of Magnetism

Magnetism is a sub-atomic phenomenon and is mainly caused due to the polarisation of electric clouds, or magnetic dipoles(and not monopoles) of certain materials with unpaired electrons. Due to this imbalance, the atom gains a net angular momentum, and a magnetic field perpendicular to the rotation of spin of the excess charge is caused. The magnitude of this magnetic or spin moment is dependent on the species of atom. This spin-spin interactions classifies materials into different magnetic classes.When atoms are brought in proximity to each other there is a probability of an electron jumping from one atom to another, known as the Heisenberg exchange. [1]. This interaction probability can indirectly couple the spin moments of the atoms, causing the spin moments to align parallel or anti-parallel. In most materials the spin moments are small and aligned randomly, leading to paramagnetism In some materials however, specifically transition metals such as nickel, cobalt, and iron, the spin moments are large, and align in parallel or ferromagnetically . This causes a net spontaneous magnetic moment in the material.

1.2 Theory of Magnetic materials and magnetism

When asked the definition of a magnet, the most probable answer is, "a magnet is a solid or a material which has a large number of atoms with magnetic moments." Magnetization M is defined as the magnetic moment per unit volume. M is considered on a length scale large enough so that one does not see the graininess due to individual atomic magnetic moments. Hence M can be considered to be a smooth vector field, which is continuous everywhere.

In free space or vacuum, the magnetic field is expressed as:

$$\overrightarrow{\mathbf{B}} = \mu_o \overrightarrow{H} \tag{1.1}$$

where, $\mu_0 = 4\pi \times 10^{-7} Hm^{-1}$is the permeability of the free space. $\overrightarrow{B}$ is simply the scaled *avataar* of $\overrightarrow{H}$ and is measured in Tesla while the former is measured in Am^{-1}.

The case is not that simple in the case of magnetic materials. The two vector fields vary differently in magnitude and direction. This classifies the material into different classes. The general vectorial relation for a magnetic material is given as:

$$\overrightarrow{\mathbf{B}} = \mu_0 \left(\overrightarrow{H} + \overrightarrow{M} \right) \tag{1.2}$$

For a linear magnetic material $\overrightarrow{\mathbf{M}} = \chi \overrightarrow{H}$and χ is called as magnetic susceptibility. Therefore the relation changes to

$$\overrightarrow{B} = \mu_{\mathbf{0}} \overrightarrow{H} \left(\mathbf{1} + \chi \right)$$

$$= \mu_0 \mu_r \overrightarrow{H} \tag{1.3}$$

where $\mu_r$is called the magnetic permeability of the material.

But what do we measure?

If a magnetic solid is placed in a free space with $\overrightarrow{B_a} = \mu_{\mathbf{0}} \overrightarrow{H_a}$ the internal fields $\overrightarrow{B_i}, \overrightarrow{H_i}$, inside the solid are not the same as $\overrightarrow{B_a}$and $\overrightarrow{H_a}$.This difference is because of demagnetization field.$\overrightarrow{B_i}$,$\overrightarrow{H_i}$both depend on the position inside the magnetic solid at which we measure them.

From Maxwell equations:

$$\overrightarrow{\nabla}.\overrightarrow{B} = \mathbf{0}$$

and since

$$\overrightarrow{B} = \mu_{\mathbf{0}} \left(\overrightarrow{H} + \overrightarrow{\mathbf{M}} \right)$$

$$\Longrightarrow \overrightarrow{\nabla}.\tilde{\mathbf{H}} = -\tilde{\nabla}.\tilde{\mathbf{M}} \tag{1.4}$$

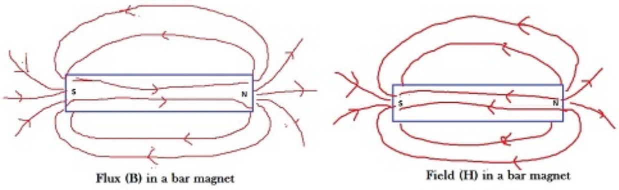


Figure 1.1: A Bar magnet

The magnetic energy is given as

$$U_m = -\frac{1}{2}\int dv\overrightarrow{\mathbf{M}}.\overrightarrow{H} \tag{1.5}$$

or in simple words, the North pole acts as a source and south pole acts as a sink.

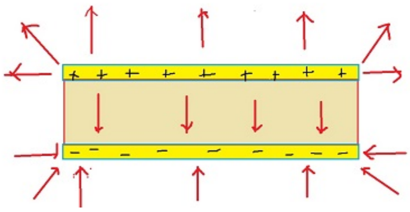


Figure 1.2: Poles of magnet

The flux closing path of least energy between the North pole and south pole is through the material, and in the inside of the material $\vec{\mathbf{H}}$ is opposed by $\tilde{\mathbf{B}}$. So, $\tilde{\mathbf{H_d}}$ is opposed to $\vec{\mathbf{M}}$ that created the poles in the first place.

So,

$$\overrightarrow{H_i} = \tilde{\mathbf{H_{ext}}} + \overrightarrow{\mathbf{H_d}}$$

taking

$$\overrightarrow{\mathbf{H_d}} = -D\overrightarrow{\mathbf{M}}$$

where D is a constant of proportionality, we get the relation

$$\overrightarrow{H_{i,x}} = \overrightarrow{H_{ext,x}} - \mathbf{D_x}\overrightarrow{M_x} \tag{1.6}$$

$\overrightarrow{H_d}$ lowers the intrinsic χ due to small length scale of the material.

$$\frac{\overrightarrow{M}}{\overrightarrow{H_{ext}}} = \chi_{apparent} = \frac{\chi_{actual}}{1 + D\chi_{actual}} \tag{1.7}$$

here D is the Demagnetizing factor.

Sample geometry	Demagnetizing factor
Flat sheet, $\overrightarrow{M}$normal to surface	1
Flat sheet,$\overrightarrow{M}$in plane	0
Cylinder, $\overrightarrow{M}$ along length	0
Cylinder, $\overrightarrow{M}$ along width	$\frac{1}{2}$
Sphere, $\overrightarrow{M}$ along orthogonal axes	$\frac{1}{3}$

Table 1.1: Demagnetizing factor. Ref. [1]

1.3 Classification of magnetic materials

Criteria for classifying materials

Magnetic susceptibility and its dependence on temperature

Susceptibility is the most important property of a magnetic material. So, the materials are divided into two broad categories. The first with $|\chi| \ll 1$, which do not have any magnetic properties and the second with $|\chi| \gg 1$, which have got colossal applications. For more concise understanding, materials are classified as χ positive or (and) χ negative.

But how is χ dependent on temperature ?

Answer : From definition, χ is the ratio of magnetization, i.e. magnetic moment per unit volume, to the applied field. If there is a change in temperature, it then affects the volume of the material and hence its magnetic moment.

For materials with positive χ, the magnitude of the susceptibility varies over a wide range. Experimentally, it is found that all these materials follow the relation

$$\chi = \frac{C}{T \pm \Theta} \tag{1.8}$$

for sufficiently high T. In this equation C, $\Theta$are positive constants and are specific signature properties of materials.

Materials with $\Theta = 0$ are called *paramagnetic* materials. Materials with negative χ are called *diamagnetic* .

Critical temperature If in the equation number 1.8 only negative sign in the denominator is considered, then.$\chi = \frac{C}{T - \Theta}$.

At T = Θ, χ will be infinite. Infinite susceptibility means that even if there is no magnetic field, there is till some finite magnetization in the material.

This kind of materials are categorized into (1) *Ferromagnetic* (2) *Ferrimagnetic*

Another class of material, with small, positive susceptibilities at all *temperatures*are called as *antiferromagnetic.* They also satisfy equation (1.8) with positive Θ and at high temperatures.

Class	Critical temperature	Magnitude of χ	Temperature variation of χ	Spontaneous magnetization	Atomic scale structure	Example
Diamagnetic	None	-$10^{-6}$to -10^{-5}	Constant	None	Atoms have no permanent dipole moment	Inert gas, Cu, Hg Bi, H_2O
Paramagnetic	None	+$10^{-5}$to +10^{-3}	$\chi = \frac{C}{T}$	None	Atoms have permanent dipole moment. Neighboring moments do not interact with one another	Cr, Mn, NO,O_2
Ferromagnetic	Curie temperature Θ_c	Large (below Θ_c)	Above θ_c $\chi = \frac{C}{T-\Theta}$ with $\Theta \approx \Theta_c$	Below Θ_c, $\frac{M_s(T)}{M_s(0)}$ against $\frac{T}{\Theta_c}$ follws a universal curve. Above Θ_c , None	Atoms have permanent dipole moments. Interaction produces $\uparrow\uparrow$ alignment.	Fe, Co,Ni
Antiferromagnetic	Neel temperature Θ_N	As paramagnetic	Above Θ_N,$\chi = \frac{C}{T \pm \Theta}$with $\Theta \neq \Theta_N$.Below Θ_N,χdecreases	None	Atoms have permanet dipole moments. Interaction produces $\uparrow\downarrow$alignment.	MnO, NiO,Cr_2O_3
Ferrimagnetic	Curie temperature Θ_c	As ferromagnetic	Above Θ_c, $\chi \approx \frac{C}{T \pm \Theta}$ with $\Theta \neq \Theta_c$.	Below Θ_c,$does$not follow universal curve. Above Θ_c, none	Atoms have permanent dipole moment. Interaction produces $\uparrow\downarrow$alignment but moments are unequal.	Fe_3O_4(magnetite)

Table 1.2: Classification of materials according to magnetic properties Ref:[1]

1.4 Motivation for the project

VibratingSample Magnetometer

The measurement of magnetic moment in magnetic materials is a widespread area of research in both academic and industrial frontier.Most common methods of magnetization measurement [2] are force method, induction method, indirect method. In force method, a magnetic dipole is placed in a field and the applied force is measured. Induction method uses the idea of relative motion between magnetic sample and detection coils. The force method though has been used of late suffers with the disadvantages of magnetization in truly uniform magnetic fields. By convention, in induction method the sample or the detection coil is passed through a magnetic pole and induced voltage is measured [3].

By designing this vibrating sample magnetometer, we have been able to measure magnetization at room temperature. The instrument was calibrated against Nickel and some other measurements of Iron, Cobalt ferrite and nickel of different dimensions were taken successfully. This shows the usefulness of the project which can be used for lab and teaching lab purposes.

The present design of a vibrating sample magnetometer employs the principle of harmonic vibration of a magnetic sample in a magnetic field. The harmonicity is achieved by employing a speaker of 4 ohm impedance. The electromagnet is driven by a lab view interfaced power supply (Kepko model).The sensing coils which remain fixed with the magnetic poles, pick-up the generated emf. The emf is locked using a Lock in Amplifier (Stanford model # SR830).

Sensitivity of the instrument was the main objective of design, and hence different combinations of coil designs were employed during the designing of the instrument. The coil were designeded keeping in mind the factors of effective sensitivity, flux detection and capture, resistance, and also sample space.

Chapter 2

Measurement of Magnetization

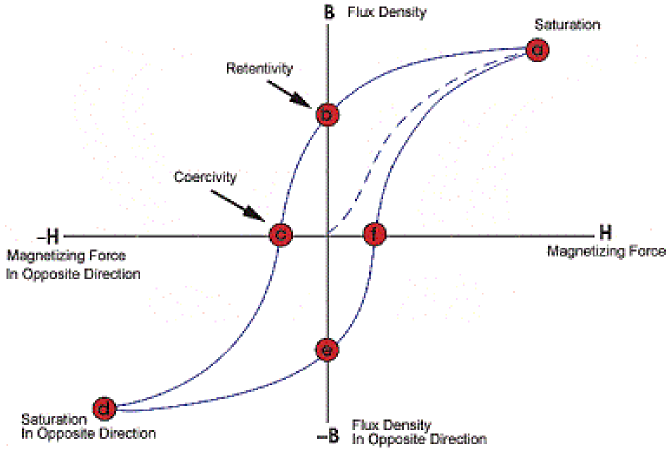


Figure 2.1: Hysteresis loop of a magnetic material

2.1 Foundations

It is known that the characterstic parametrer that describes a material to be magnetic is its hysteresis loop. The measurement of the hysteresis loop, i.e. magnetization as a function of applied field gives us the idea about the usefullness of the material. From these measurements, we can easily deduce the values of saturation magnetization, coercivity, remnance, permeability and susceptibility. We can also know the variation of magnetization with temperature.

In this chapter, the various methods to measure magnetization will be described.

2.2 Methods.

The measurement of magnetic moment or magnetization of different magnetic materials has been a widespread area of research in both academic and industrial institutes. The most common methods of measurement of bulk magnetization

(1) Force method

The force experienced by a magnetic dipole which is placed in a magnetic field gradient is measured. The magnetization can hence be calculated using Faraday's law.

(2) Induction method

The magnetic dipole has a relative dispalcement with respect to detection coils. The voltage due to induction can be used to measure magnetization.

(3) Indirect methods

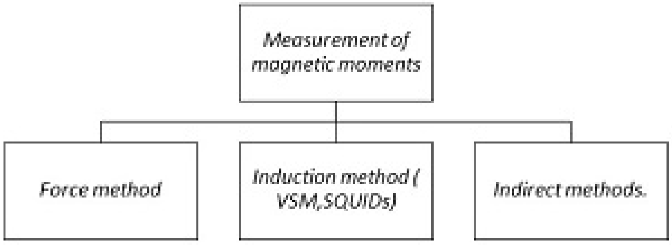


Figure 2.2: Measurement of Bulk magnetization

2.2.1 Force method.

In this method, magnetization is measured by measuring the force exerted on a specimen by a magnetic field.

When a magnetic dipole of moment m is placed in a magnetic field $\mathbf{H}$, the energy changes by an amount

$$E = -\frac{1}{2}\mu_0 m\mathbf{H} \tag{2.1}$$

Since the magnetization $\mathbf{M}$ is given as $\mathbf{M} = \frac{\mathbf{m}}{\mathbf{V}}$, where V is the volume of the specimen.

$$\Rightarrow E = -\frac{1}{2}\mu_0 \mathbf{M}V\mathbf{H} \tag{2.2}$$

If the energy changes only in the x direction,

$$F = -\frac{\partial E}{\partial x} = \frac{1}{2}\mu_0 V\frac{\partial\left(\mathbf{MH}\right)}{\partial x} \tag{2.3}$$

For a ferromagnetic material, it can be assumed that,

$$\mathbf{M}\left(\mathbf{x}\right) \simeq \mathbf{M}$$

so we get,

$$\boxed{F = \frac{1}{2}\mu_0 V\mathbf{M}\frac{\partial\left(\mathbf{H}\right)}{\partial x}} \tag{2.4}$$

This means that, if a magnetic material is placed in a non-uniform field $\mathbf{H}$ it will experience a force given by the equation 12.

The specimen is suspended from a sensitive balance and the change in its *apparent* weight is measured when the magnetic field is switched on or off. This method is called the Faraday's method. [4]

Force method is a sensitive technique, but suffers with the following disadvantages:

(a) Difficult to measure magnetization in truly uniform magnetic field.

(b) Not easily adaptable to routine measurements of magnetization versus applied field or crystallographic orientation.

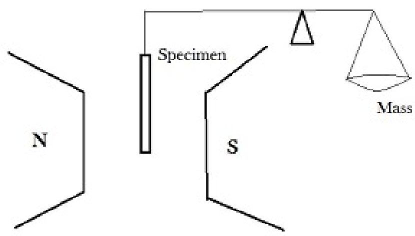


Figure 2.3: Faraday's Balance

2.2.2 Induction method.

This method uses the principle of Faraday's laws of electromagnetic induction. To illustrate this method, a coil configuration is shown below:

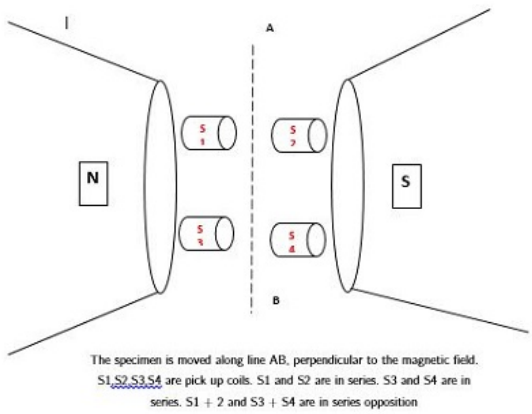


Figure 2.4: Induction method

The magnetic moment of the specimen, induces EMFs opposite spin inn S1+S2 and S3+ S4. Since the coils are connected in series opposition, the signals are added up.

The signal produced in the coil is thus proportional to the magnetic moment of the specimen.

This method is used in VSM, SQUID magnetometers.

2.2.3 Magnetometry by means of Hall method.

Hall effect is the appearance of transverse voltage or Hall voltage (V_H) when a chrged conductor is placed in a perpendiclaur magnetic field.

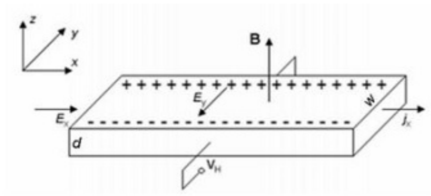


FIG. 1: Schematic view of Hall's experiment

Figure 2.5: Hall measurement

As a result;

$$V_H = \frac{R_H I \mathbf{B_z}}{d} \tag{2.5}$$

where I is the driving current, $\mathbf{B_z}$ is the magnetic field along z-axis and d is the thickness of the film, and R$_H$is the Hall coefficient.

Since

$$R_H = \frac{1}{ne}$$

n being the charge carrier density and e=-1.6$\times$10^{-19}C.

Thus using this tecnique, magnetic field can be easily measured.

2.2.4 Indirect methods

There are other methods which employ imaging techniques to find magnetization. One such technique is MOKE or *Magneto Optic Kerr Effect*. Magneto optical effects in magnetic materials are due to anisotropy of materials. The source of this optical anisotropy is the magnetization **M** within surface domains, which can be influenced by extrenal forces such as magnetic fields. the optical anisotropy alters the state of linearly polarised light which is reflected off the magnetic film

Chapter 3

Designing of the Vibrating Sample Magnetometer

Introduction

The Vibrating Sample Magnetometer is a sensitive and versatile instrument for study of magnetic moments in different magnetic materials as a function of magnetic field and,/ or temperature. Its use to measure magnetization is based on the Faraday's laws of electromagnetic induction.

A system to work up to 0.6 Tesla with temperature range of 100 K to 400 K is attempted. The system designed has been used to measure the magnetization of elemental ferromagnetic materials like Fe, Ni of different dimensions as well as composites of Barium, Cobalt, and Lanthanium.

In this chapter, the following sections will be discussed;

1. Theory of working of VSM
2. Details of the constituent components
3. Detailed construction design of the instrument.

The measurement and callibration subjects will be discussed in the next chapter.

3.1 Theory of working of VSM

The VSM was first designed by Simon Foner, in 1959 at the Lincoln laboratories. [2]. Since then many transformations and variants have been seen [3], but the the basic underlying principle remains the same- induction of *emf* by Faraday's law.

All the VSM work, involves the measurement of voltage induced in a stationary coil, otherwise called as detection coils, due to the harmonic vibration of the sample in a uniform magnetic field.

It is thus the detection coil geometry and configuration which decides the sensitivity of measurement.

By employing electromagnetism principles, the induced voltage in the detection coil can be calculated.

Suppose due to vibration of the sample, a *flux* ϕ is picked up by the detection coil.

So,

$$\phi = \mu\mu_0 H$$

where, $\mu_0 = 4\pi\times10^{-7}$

μ= Point dipole and H is the magnetic field.

Due to the vibration, the flux linked to the coil changes and a voltage is induced.

$$V(t) = -N\frac{\partial\phi(t)}{\partial t}$$

here N= number of turns in the detection coil.

or,

$$V(t) = -N\mu_{\mathrm{o}}[\left(\frac{\partial H_x}{\partial x}.\frac{\partial x}{\partial t} + \frac{\partial H_x}{\partial y}.\frac{\partial y}{\partial t} + \frac{\partial H_x}{\partial z}.\frac{\partial z}{\partial t}\right)\mu_x +$$

$$+\left(\frac{\partial H_y}{\partial x}.\frac{\partial x}{\partial t} + \frac{\partial H_y}{\partial y}.\frac{\partial y}{\partial t} + \frac{\partial H_y}{\partial z}.\frac{\partial z}{\partial t}\right)\mu_y + \left(\frac{\partial H_z}{\partial x}.\frac{\partial x}{\partial t} + \frac{\partial H_z}{\partial y}.\frac{\partial y}{\partial t} + \frac{\partial H_z}{\partial z}.\frac{\partial z}{\partial t}\right)\mu_z]$$

From this it can be deduced that the induced voltage depends on:

1. Number of turns
2. gradient of H .

If the vibration is harmonic in z axis.

$$z\left(t\right) = z_o sin\left(\frac{vt}{z_0}\right)$$

so

$$\boxed{v\left(t\right) = -N\mu_0 v\left(\frac{\delta H_x}{\delta z}\mu_x + \frac{\delta H_y}{\delta z}\mu_y + \frac{\delta H_z}{\delta z}\mu_z\right) cos\left(\frac{vt}{z_0}\right)}$$

here, z_0= vibration amplitude, υ= velocity of the moving sample.
As per the analysis of A. Zieba [5], for a coil carrying current,

$$B.\mu = I\phi$$

$$v\left(t\right) = \frac{\partial\phi}{\partial t} = grad\left(\frac{H\left(r\right).\mu}{I}\right).v\left(t\right)$$

or

$$\boxed{v(t) = \mu G\left(r\right) v\left(t\right)}$$

The scalar $G\left(r\right)=\frac{d}{dz}\left(H(r)\,\frac{\mu}{I}\right.$ is called the sensitivity function, and represents the derivative alomg the direction of sample motion.

Therefore if the amplitude and frequency of the vibration is known, along with the sensitivity constant, the voltage induced in the detection coil is proportional to the magnetic moment and hence magnetization of sample.

The different possible coil arrangements are :

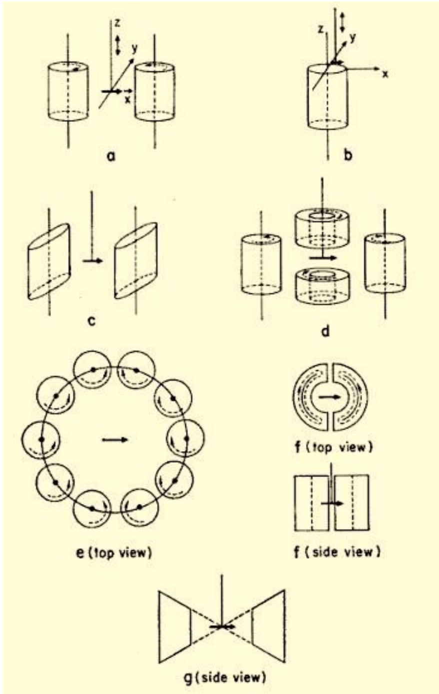


Figure 3.1: Different coil designs Ref:[2]

3.2 Details of the constituent components

The different constituent parts of the VSM. are

1. The vibrating system
2. Lock-In amplifier
3. Electromagnet and power supply
4. Pick up coil or Detection coil
5. Sample
6. Temperature variation system

3.2.1 The vibrating system

From previous discussion, it is followed that the vibration of the sample must be driven by an unit which vibrates harmonically, with low noise and without damping.

So a low frequency speaker is employed, which oscillates the sample rod in z axis.

The small amplitude of vibration is not directly measurable [6]. However the accoustic intensity is directly proportional to the square of the current through the speaker. So, the speaker current is a measure of the the vibration amplitude. The induced signal is found to vary linearly with speaker voltage and hence with the vibration amplitude.

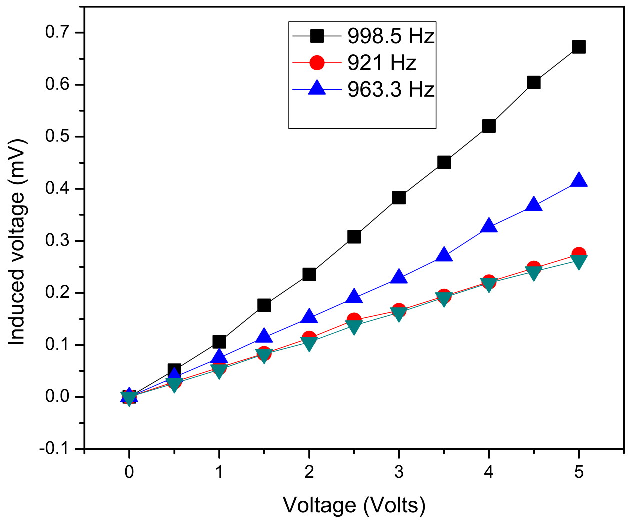


Figure 3.2: Voltage response to frequency

3.2.2 Lock-in Amplifier.

The speaker is driven by a sinosoidal output of a lock-in amplifier (Stanford model SR830 DSP Lock-in amplifier). The lock-in amplifier is also used to detect and lock the induced voltage of the detection coils.

Lock-in amplifiers are used to detect and measure very small AC signals, all the way down to nano volts [7]. These use a tecnique called as phase- sensitive detection to single out the component of the signal at a single reference frequency and phase.. Noise signals at frequencies other than the reference frequency are rejected and do not affect the measurements.

Figure 3.3: Stanford model SR830 Lock-in Amplifier

The SR830 lock-in amplifier has an inbuilt sine wave generator.. All components of the input signal are multiplied by the reference simultaneously. The product of this multiplication yields a AC output signal proportional to the components of the signal whose frequency is locked by the reference frequency.

3.2.3 The elctromagnet and power supply.

- Electromagnet

For producing the uniform magnetic field, we have used a teaching lab electromagnet (by SES Instruments model #EMU-50). The electromagnet can produce a field of 2500 gauss.

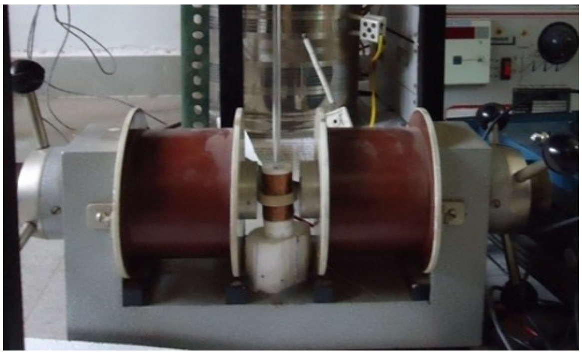


Figure 3.4: Electromagnet

- Power supply

The electromagnet is sourced by Kepco BOP 25- 40 DC bipolar power supply. The power supply is interfaced by *Labview* to allow the current sweep controlled by DAQ.

Figure 3.5: Kepko BOP 35-40 DC

3.2.4 Detection coil

Since measurement of flux is the heart of the principle of working of the VSM. Besides sample position, dimension another thing that affects the magnitude of induced voltage is the coil geometry. The parameter which describes the dependence of the output voltage on the coil and sample geometry and their relative displacement is the sensitivity function. By the principle of reciprocity [8]. " Magnetic flux produced by magnetic moment in a coil of arbitary geometry is equivalent to the field **B**(at the position of moment) produced by the same coil carrying a current I."

$$B.\mu = I\phi$$

For a transverse design or set up, in which the direction of moment vibration is perpendicular to the applied magnetic field, in which the requirement of insetivity due to sample position and geometry is to be fulfiiled: Place the sample at the saddle point of sensitivity function. ie;

at the point at which $\frac{\partial G}{\partial x} = \frac{\partial G}{\partial y} = \frac{\partial G}{\partial z} = 0$ but $G(r) \neq 0$. Therefore the various coil configurations which satisfy this saddle point situation is shown below:

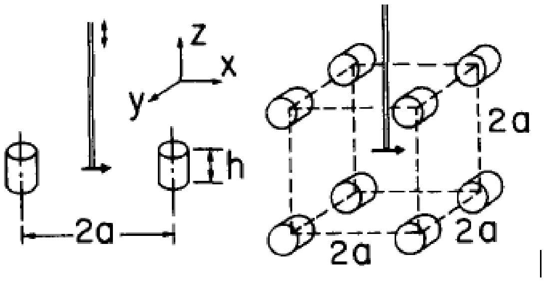


Figure 3.6: Saddle points

However be the design of the detection coil, the winding must be done in series opposite so that the induced voltage gets added up and also the effect of the extrenal magnetic field is zero.

3.2.5 Sample.

Measurements of magnetization was done on various samples, from elemental metals like Fe, Ni, to composites of La, Co, Ba etc. Pure nickel (99.99 % purity) was used for the callibration. The callibration and measurement process will be discussed in the next chapter.

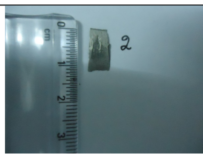
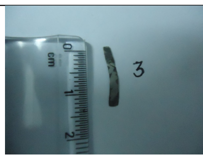
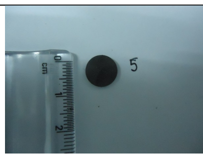
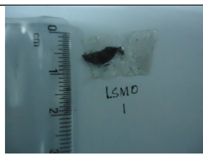
Srl. #	Sample name	Sample	Weight (mg)	Dimension(l,b,t) (mm,mm,mm)
1	Nickel (99% pure)		87.79	0.423,2.236,9.98
2	Nickel (99 % pure)		181.07	0.196,5.546,11.493
3	Nickel (99 % pure)		44.445	0.1466,1.986,14.34
4	Cobalt Ferrite		48.03	0.1457,10
5	Barium Ferrite		46.09	0.055,10
6	$La_{0.8}Sr_{0.2}MnO_3$		55.28	0.145,10

Table 3.1: Samples used

3.2.5.1 LSMO synthesis.

The variation of magnetization with temperature was studied and measured for $La_{0.8}Sr_{0.2}MnO_3$.

The synthesis of the nanoparticles of LSMO are done by using auto combustion sol gel technique. [9].

Precusors for the synthesis are:

- $La(NO_3)_3.6H_2O$
- $Sr(NO_3\ )$
- $MnNO_3$.
- Stoichiometric amounts were taken as 0.8 % of 0.01 mole,0.2 % of 0.01 mole 0.2% of 0.01 moles respectively.0.01 mole of glycine was taken as combustion agent.

- Ratio of glycine to metal nitride was 1:1.

The reactants were added to distilled water and stirred thoroughly. When the solution turned to colourless, glycine was added. The solution was then heated with constant stirring on a magnetic stirrer. After 3 hours of heating, the solution changed into a viscous gel. Then after burning due to glycine, the gel converted to a black powder . After collecting the powder, calcination was done at 600˙celcius for 2 hours. After cooling the sample was collected and grinded for 2 hours. The sample was then pressed into a pelet form and sintered at 825˙celcius for 5 hours.

3.2.6 Temperature variation.

In order to study the variation of magnetization with temperature, a simplistic method of producing temerature variation and measurement have been set up .

The cooling is done by liquid Nitrogen (100 K) and heating is acheived by resitance heating method (400 K).

3.3 Detailed construction design and working of the instrument.

The construction of the various constituent components of the VSM have been described and illustrated in this section.

3.3.1 Vibrating system.

As already mentioned before, for periodic, harmonic, low frequency, longitudinal vibration, a mid range audio speaker of 4 Ω impedence was chosen. The normal output of the firstspeaker used was 5 W and that of the second speaker was 80 W.

A *hylam* cap was glued to the diaphragm of the speaker . Precautions were taken to prevent the cone to stick and hus prevent vibration.An acrylic rod was then glued to the open space of the cap with additional covering support. Care was taken to ensure the linearity of speaker's performance by constant vibration amplitude.

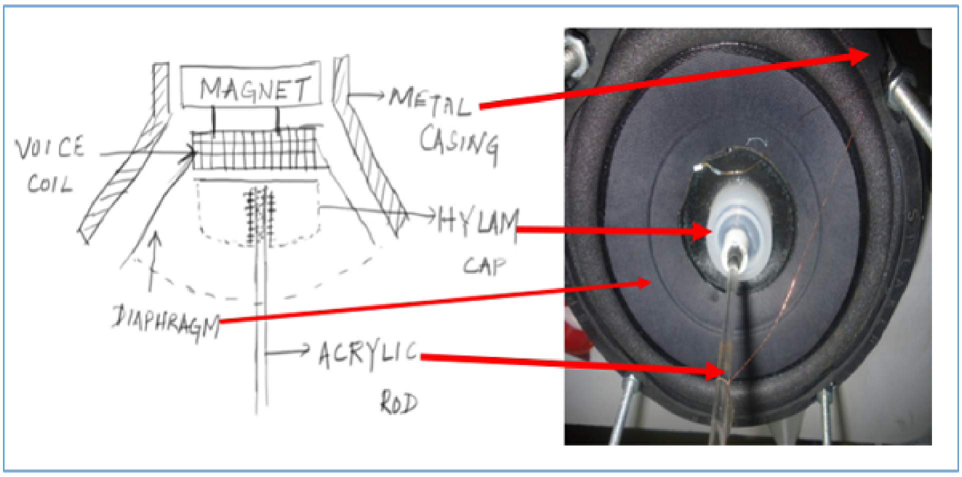


Figure 3.8: Vibrating system

The speaker assembly was firmly screwed and to a wooden base by screws, such that the speaker was placed 70 mm above the base. To prevent the vibration to be transmitted into the lower end of the instrument and thus preventing noise, thick rubber bushes were coupled with the screws. Additional sponging material was laid below the wooden frame.

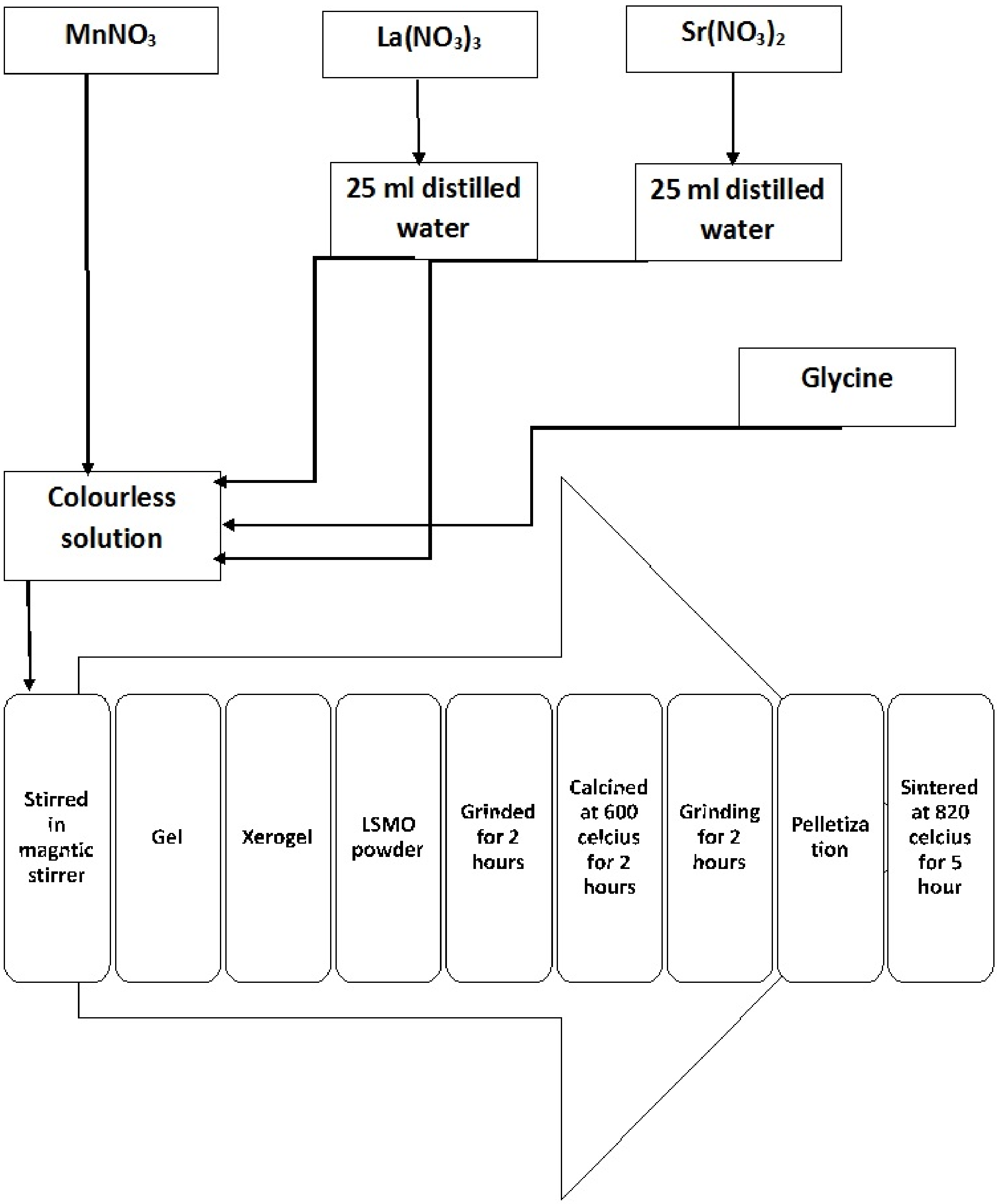


Figure 3.7: Synthesis of LSMO using sol- gel technique

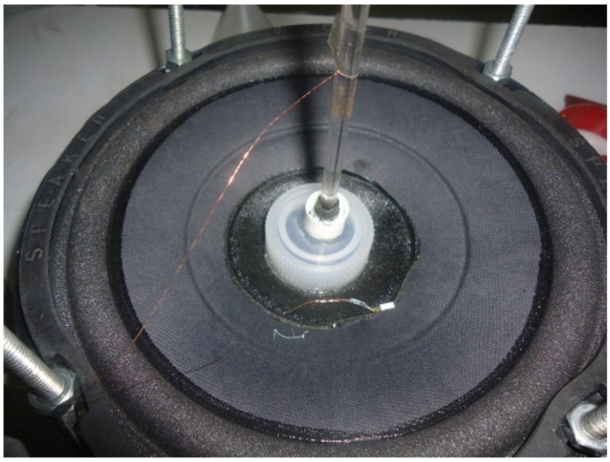


Figure 3.9: Speaker with the sample rod

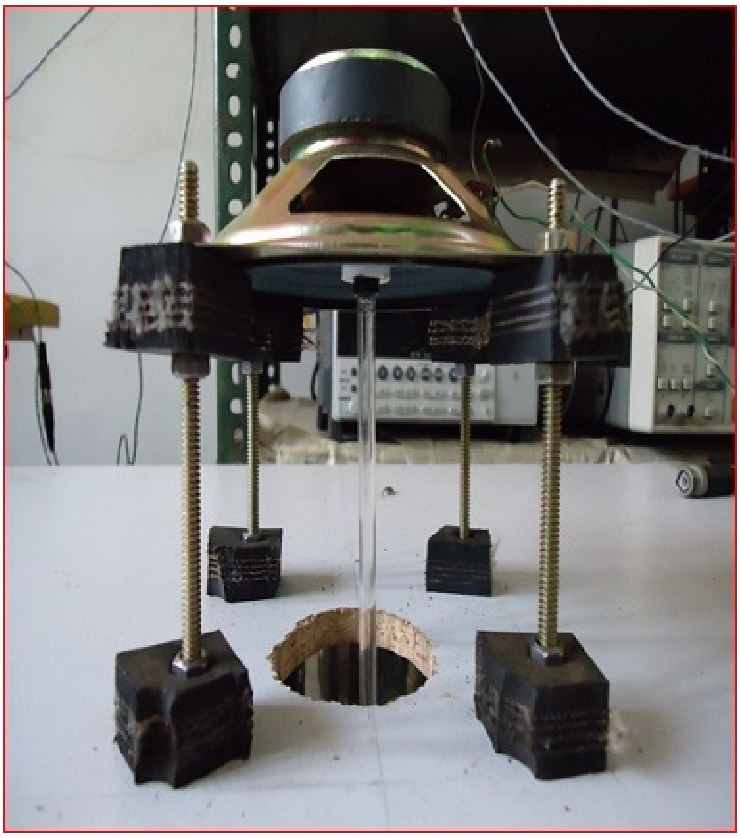


Figure 3.10: Arrangement of the initial speaker

3.3.2 Magnetic field.

The electromagnet was driven by *Kekpko* power supply. The variation of magnetic field with input current was measured using a gaussmeter.

The probe of the gauss meter was placed between the poles of the magnet, while the input current was incremented. The field versus current data was then tabulated for necessary callibration.

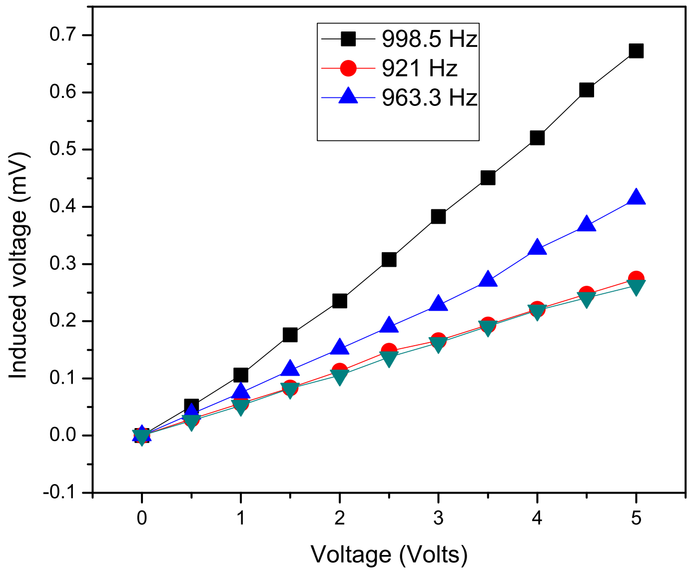


Figure 3.11: Variation of magnetic field with current

3.3.3 Detection coil .

Enamled copper coils were used to wind a number of designs. These coils were in series opposite resistance so that the effect of the extrenal field is cancelled out and also the induced voltage is added up. The detection coils were designed keeping in mind the parameters like sensitivity, resistance, effective sheilding, and availaibility of sample space . The different coil designs incorporated for the design of the VSM are:

3.3.3.1 Design # 1:

In this design, a hylam rod of 26 mm diameter was chosen as a firmer. The rod was machined as per the dimension shown below. The required space was then winded using enamled copper wire of 36 AGW. Each space had 500 turns, so in total there were 1000 turns. The winding were done in series opposite. The middle portion of the coil was a cavity through which the sample was made to vibrate freely. The sample was placed in between the two coils.

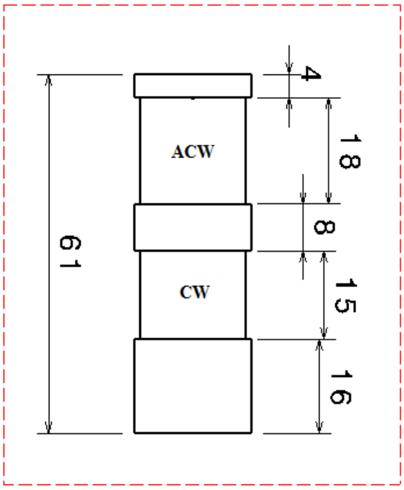


Figure 3.12: Schematic of Coil design 1

Figure 3.13: Coil design 1

3.3.3.2 Design # 2:

In this design, used pen refills of 2 mm diameter was used as former for coil winding. Refills were cut to 6 cm length each, and 4 such arrangements were made.. To the ends of the refill. thick tapes were put to give support to the copper wires. Each of the refill were then winded using coils of 36 AGW. In total there were 2400 turns in the entire arrangement of 4 coils. The coils were then held on a base , with coil separation as given. The ends of the wire where then joined as per series opposite winding, with two ends for output signal. Soft iron nails were introduced into the cavity of the refills to improve the sensitivity of the induced signal.

Coil #	1	2	3	4
Polarity(end-end)	(+ -)	(- +)	(+ -)	(+ -)

Table 3.2: 4 coil winding

Figure 3.14: Design no 2

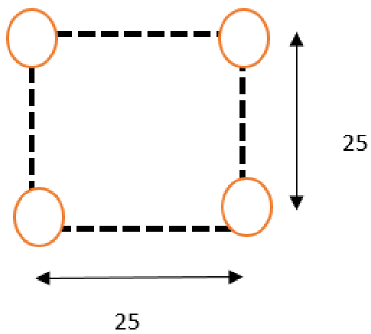


Figure 3.15: Design 2 coil arrangement

3.3.3.3 Design # 3:

In this design the 4 coil arrangement was improvised further to give an 8 coil arrangement, aiming the sensitiity and the effect of sample position on induced voltage signal. For the construction of this set up, hylam rod of 6 mm diameter was chosen as firmer. Each branch of the coil design was designed as shown in figure below:

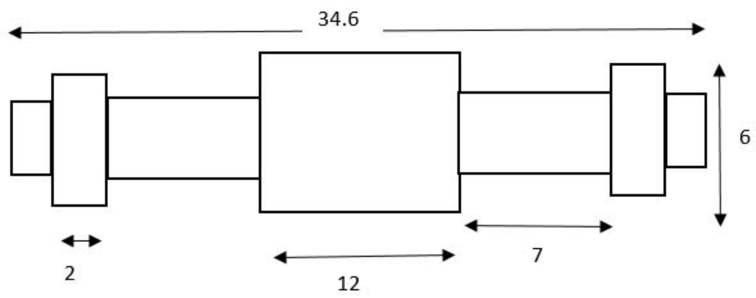


Figure 3.16: Schematic of 8 coil arrangement

280 windings were done to each small space availaible. Thus in total there were 2240 number of turns in this set up.

The coils were connected in the pattern as shown in the table:

Coil #	1	2	3	4	5	6	7	8
M_x	-	-	+	+	-	-	+	+
M_y	-	+	+	-	+	-	-	+

Table 3.3: 8 coil system

The schematic of the coil and the final coil design is shown here:

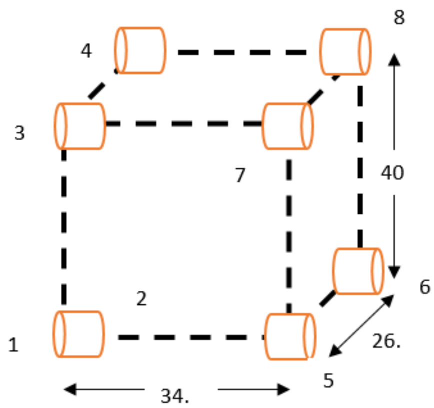


Figure 3.17: Schematic of 8 coil system

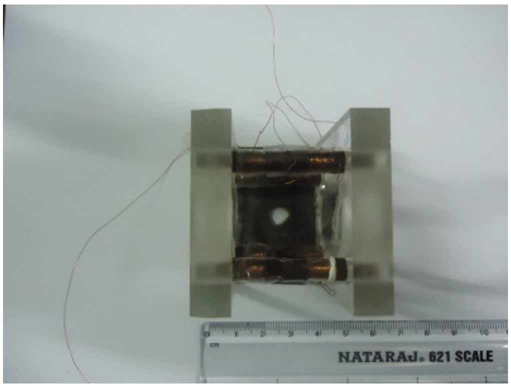


Figure 3.18: 8 coil system detection coils

3.3.3.4 Design # 4:

For the final design of the pick up coils , the relay coils were used. Two relays were opened up and the coil in them (about 3200 turns each) were used. The soft iron sheet at the back bone of the coil was also removed. Instead soft iron nails were introduced into the cavity to improve sensitivity of the coils. The coils were then glued to the poles of the electromagnet, Thus using this design we could play around with the distance between the magnetic sample and the coils. Besides giving a good induced signal, space was also acheived to install other temperature variation set ups.

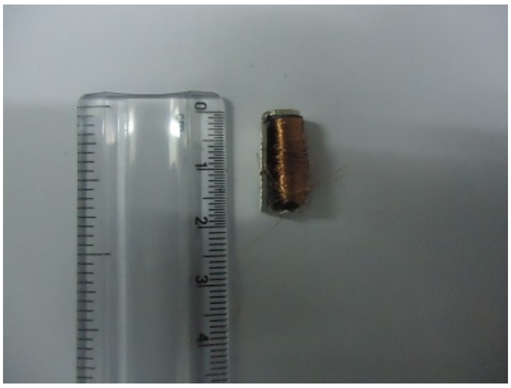


Figure 3.19: Design 4 of coil design

3.3.4 Temperature variation.

For measurement of magnetic field as a variation of temperature, it was necessay

- to produce temperature variation
- measure the temperature.

3.3.4.1 To produce temperature variation.

Nichrome wire of 20 Ω resistance was wound around a test tube which was then co-axially fitted to a plasic container. Resistance heating was done by passing current to the nichrome wire.

For lowering the temperature, the outer plastic container was filled with liquid nitrogen.

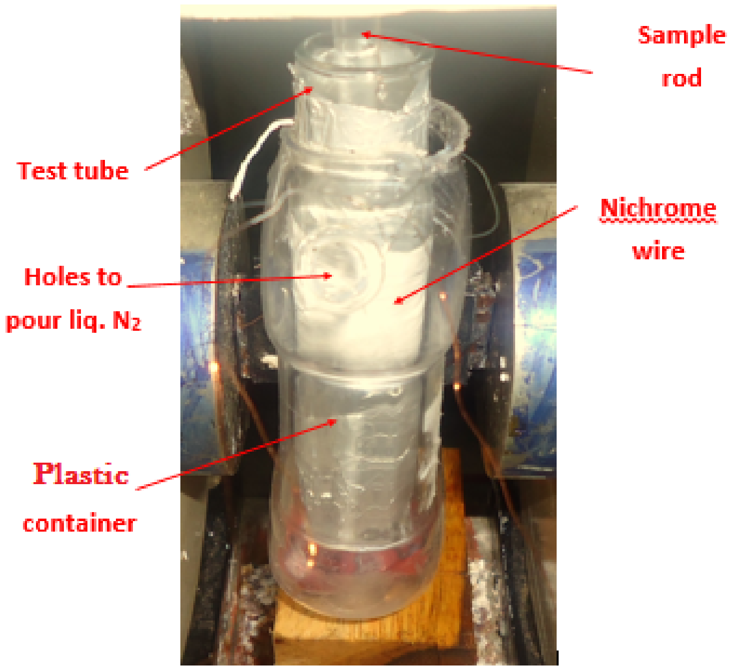


Figure 3.20: Temperature variation set up

3.3.4.2 Measurement of temperature.

To measure the temperature, a PT 100 sesnsor was firmly attached to the sample rod and its ends were connected to a multimeter. The resistance values were tabulated .

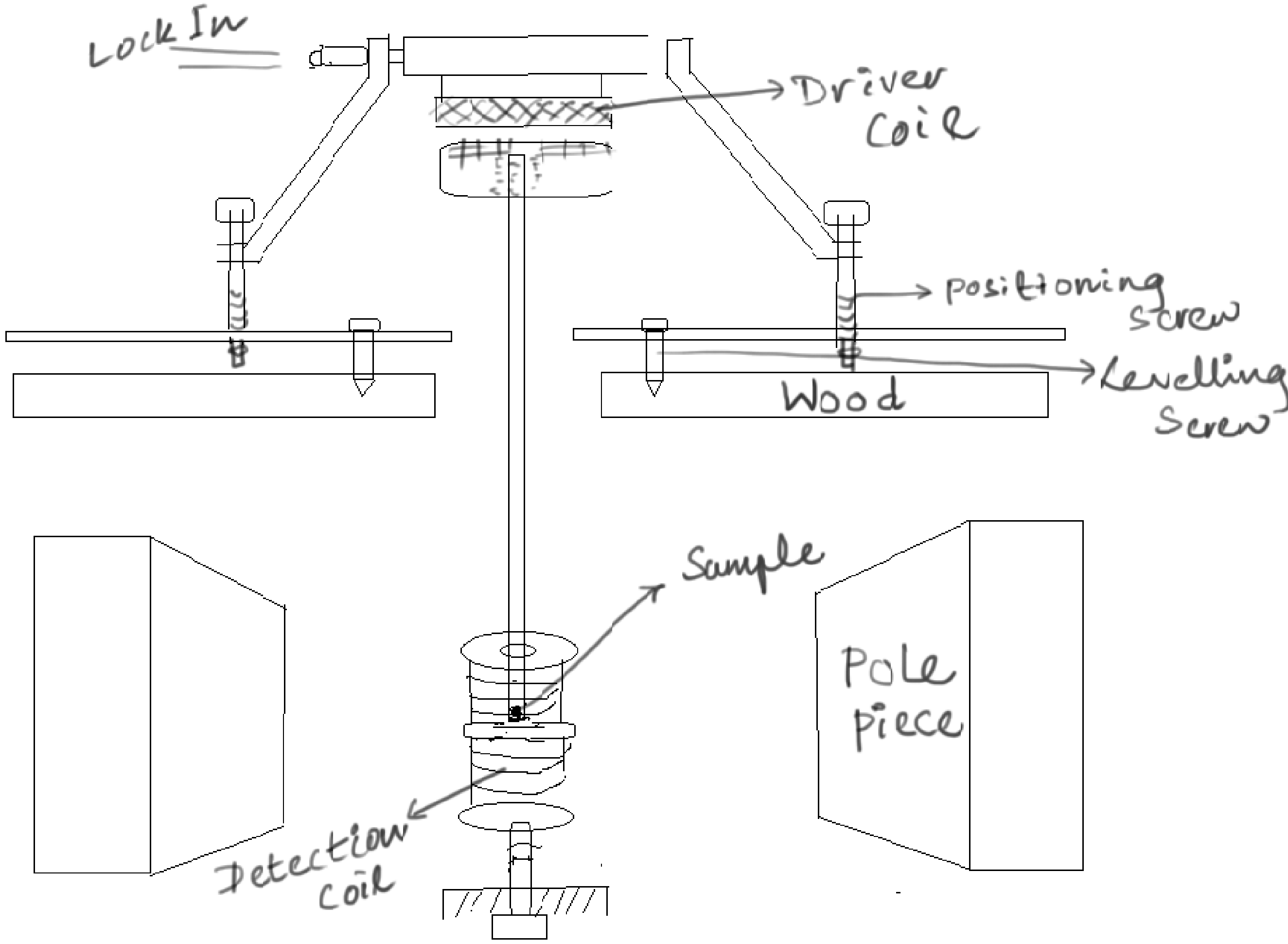


Figure 3.21: Schematic of the VSM

The general schematic of the VSM is shown :

Chapter 4

Measurement methodology and Results.

4.1 Measurement scheme.

This was the final and most important step of the experimental set-up.

For making the measurements and the behaviour of the VSM automated, that is; frequency sweep, voltage/ current ramping rate, magnetic field sweep, temperature sweep, etc , home made programs and DAQ assistants were developed using *Labview* .

The DAQ is responsible for complete automation and control, invoking input parameters, response to the input parametrs, recording the behaviour, and displaying the output to the user.

The mechanism of measurement is shown in the schematic below.

1. The user inputs the necessary working parameter like amplitude, frequency, sweep rate in the *Labview* DAQ.

2. After receiving the input arguements, the DAQ directs the power supply and the Lock-in Amplifier .

3. The power supply drives the electromagnet to provide the necessary magnetic field and its ramp rate.

4. The lock in amplifier drives the speaker to its vibration harmonically between the coils.

5. The induced voltage in the detection coil is then locked by the lock-in amplifier.

6. The locked voltage signal is then sent via GPIB to the labview DAQ.

7. The DAQ gives the desired output to the user.

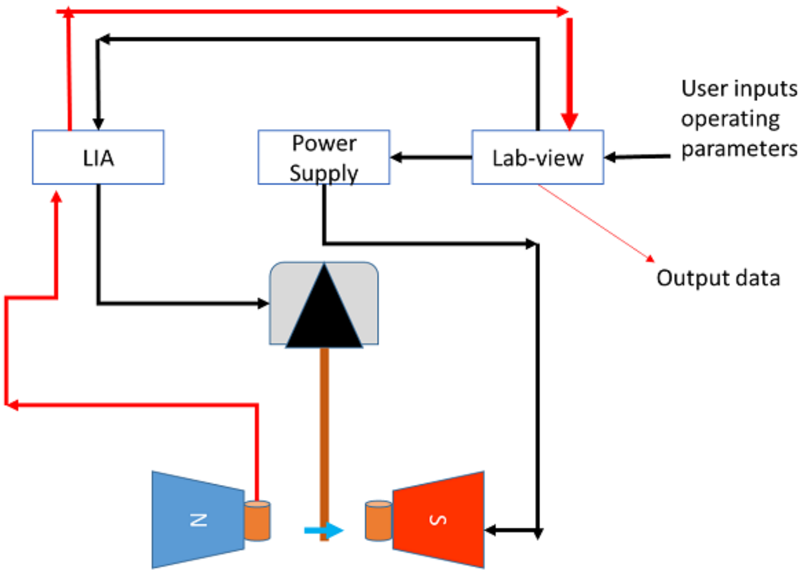


Figure 4.1: Measurement flow-scheme

4.2 Measurement.

Before the instrument was prograamed to give us magnetization results, the first step that was checked whether we were able to lock signal. And how does the signal vary with the step/ ramp rate and with sample to sample aand at differenet frequencies and amplitude.

4.2.1 The magnetic field.

A probe of a gaussmeter was placed between the poles of the electromagnet, and the input current was changed from maximum to minimum value using the DAQ.

The magnetic field recorded in the gaussmeter was tabulated against the input current.

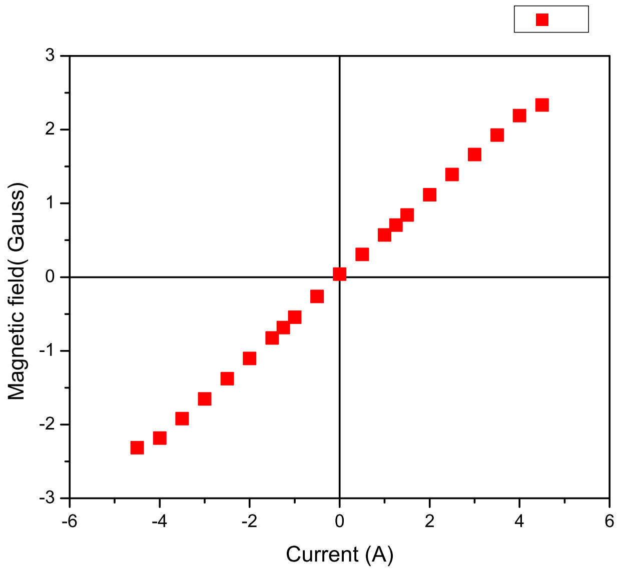


Figure 4.2: Variation of magnetic field with current

For the measurements of magnetization in later part of the project, the current was converted to magnetic field using this data.

4.2.2 Signal and frequency selection.

The nature of signal induced in the detection coil as a function of amplitude and frequency of driving signal fed by the lock-in amplifier was taken.

The graphs shown in Fig 4.3 are the variation for the coil # 1. The sample was placed in between the two coils.

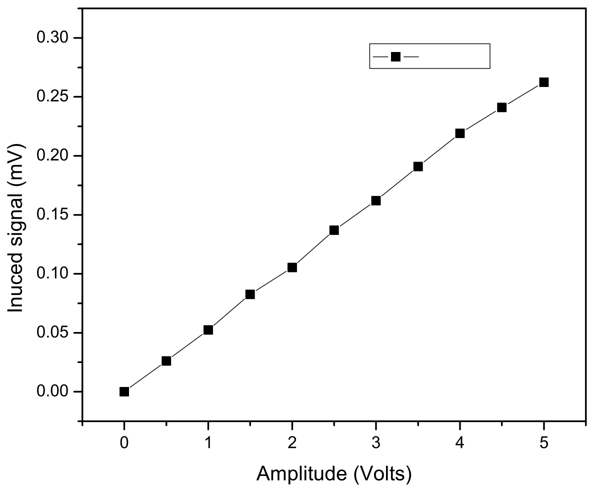
The linearity of the signal depicted the linear response of the einduced signal with vibrating amplitude. This method was used to measure the vibration amplitude of the speaker.

The next step was to find the frequency for which we get the maximum signal and free from noise. The frequency variation was done for the coil # 3 at 5 volts, 4 volts, and 3 volts amplitude voltage for different ferromagnetic materials like Nickel , Iron.

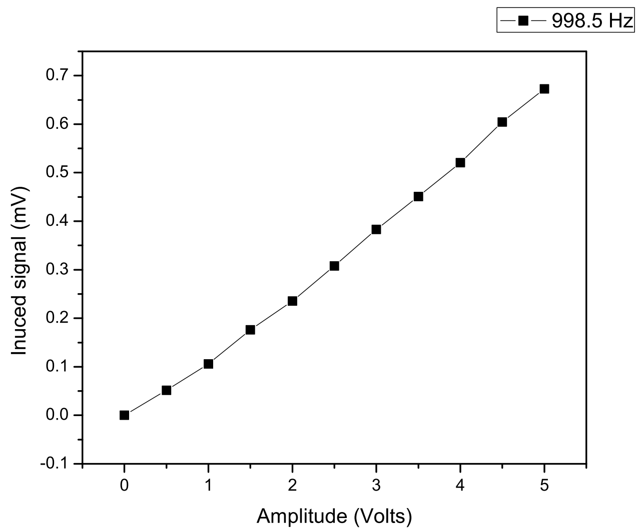
From the induced signal data, we found out that the maximum signal was locked at the frequency bands of:

1. 60 Hz to 95 Hz

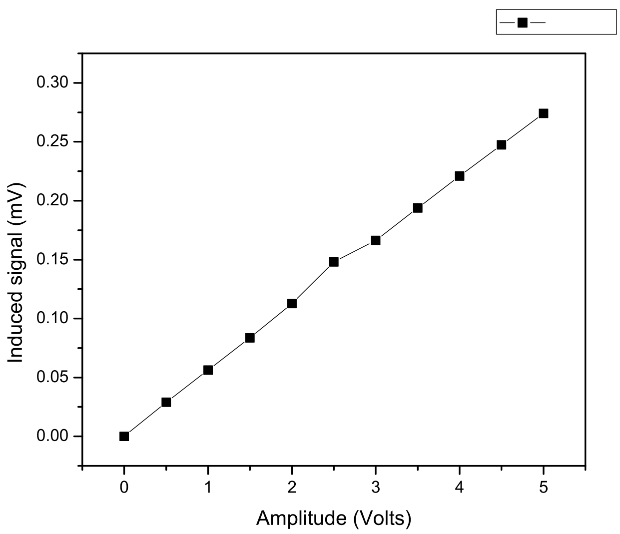
2. 110 Hz to 140 Hz



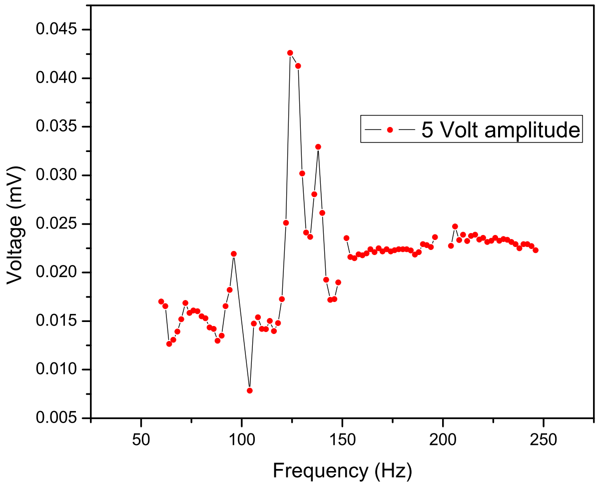
(a) Frequency = 849 Hz



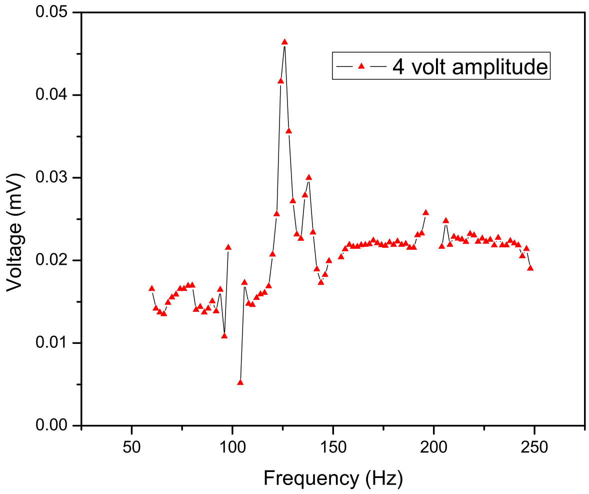
(b) Frequency = 998.5 Hz



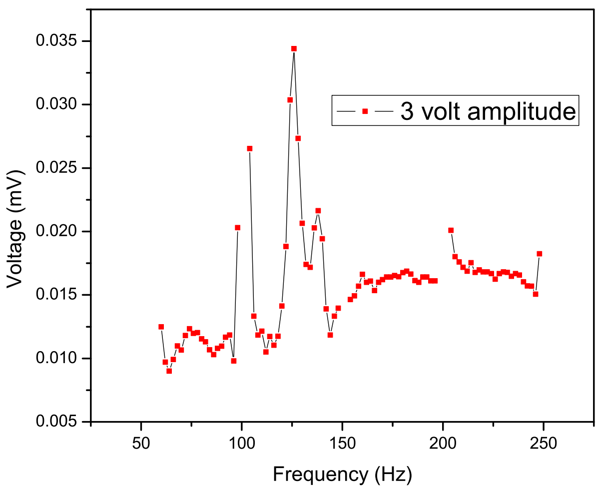
(c) Frequency = 921 Hz



(a) Amplitude 5 volts



(b) Amplide 4 volts



(c) Amplitude 3 volts

3. 165 to 175 Hz
4. 210 Hz to 250 Hz
5. and at higher frequencies above 600 Hz.

For linearity of signal and a safe limit from line frequecy of 50 Hz, the region between 110 Hz to 140 Hz was chosen as the working input frequency of the VSM.

4.2.3 Measurements

Here is the compilation of magnetic measurements for the coil designs 2, 3 and 4. The coil design number 1 was not used , because of damping of the signal.

.

Reasons for not using the coil design # 1:

- Bulky, leaving no space for additional room for other design between the poles of the magnet.
- Difficulty in placing the sample exactly in the middle of two coils. If the experiment is repeated, the sample position was not reliable.
- Difficulty during mounting sample. Had to remove the vibrating unit everytime new sample was to be mounted.
- Damping of vibration. At high magnetic field the sample stuck to the inner side of the hylab tube, thus damping its vibration and giving noisy data. There were no provisions to guide the direction of vibration.
- Shielding of the low induced signal due to thick walls of the firmer.
- Ratio of winding area to sample vibration was too high, thus the sensitivy was also not good.

4.2.3.1 Coil design 2.

The four coils were placed between the poles of the magnet as discussed earlier. The measurement conditions were:

- Frequency of input signal= 134 Hz.
- Amplitude of input signal= 5 volts (p-p).
- Time constant = 3 $\times$100 ms
- Time delay = 3 seconds.

- Sample chosen Nickel (Srl # 1 of Table 3.1)

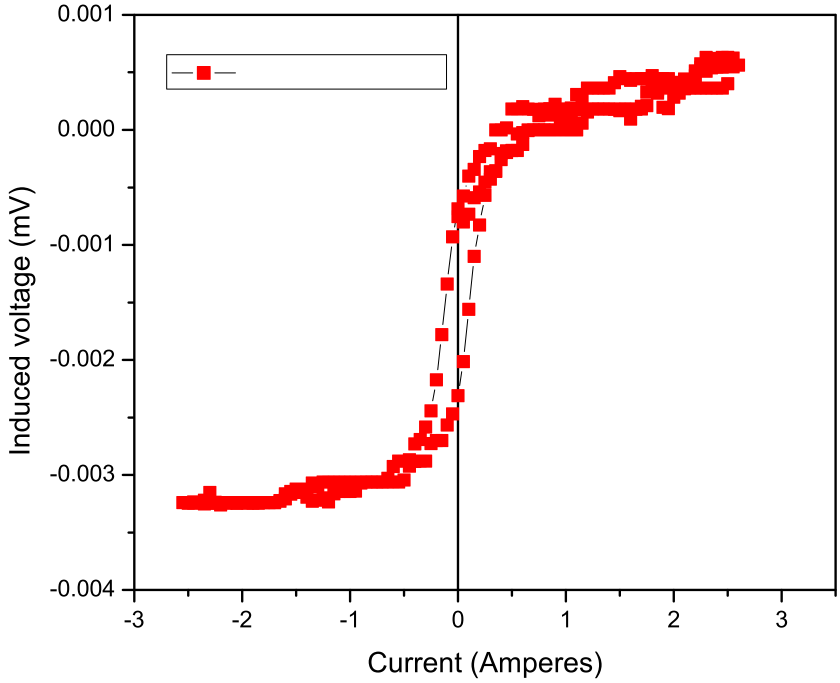


Figure 4.5: Hysteresis loop from coil 2 of Nickel sample

From the graph it is evident that the induced signal was very weak. So the trail was measured again with introducing small stainless steel pins to the cavity of the refills.

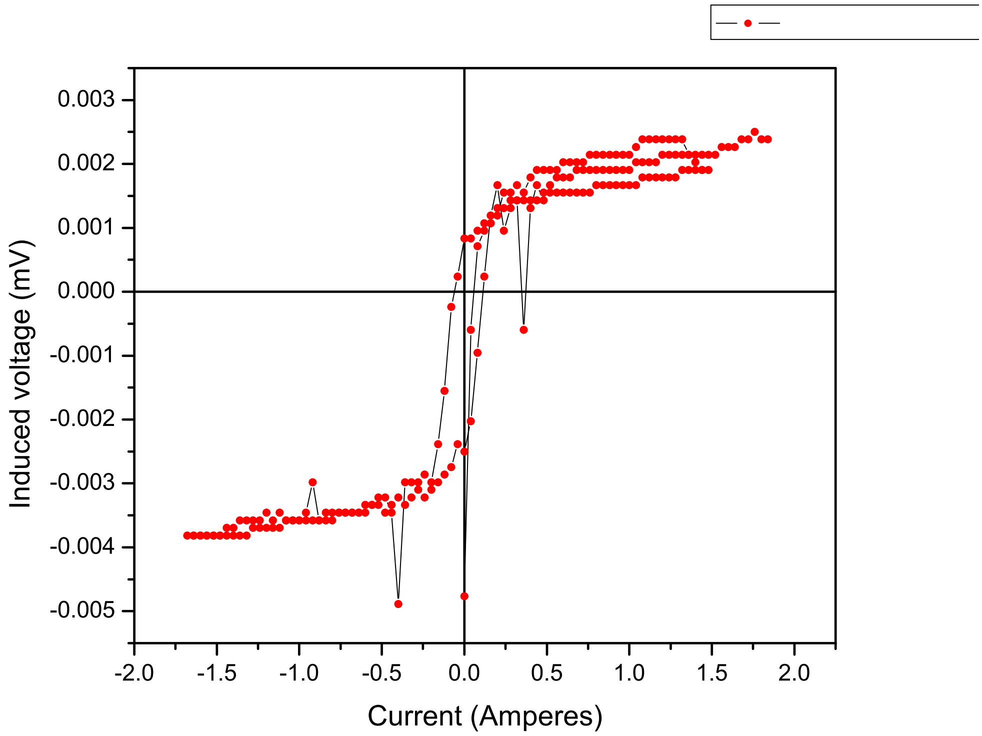


Figure 4.6: Hysteresis loop from coil 2 of Nickel sample with pins

From the results obtained it was conceived that.
Sucess

- Hysteresis loops were obtained for the sample.
- Coil worked for other samples like iron, nickel of smaller volume.
- No damping of vibration of sample, and no extrenal guide was required.
- Enough room to install temperature variation set up.

Failures

- Voltage induced was very low.
- Introducing soft iron core, improved the sensitivity a bit. But as they were mobile, at high fields interferred with signal and also gave noisy data.
- Difficulty in sample mounting. Had to remove the coil each time, when sample was to be mounted, thus the position got varied.
- Effect od sample geometry was prominent.

4.2.3.2 Coil design 3.

The coil design 3 was placed between the poles of the electromagnet. The sample rod was placed at the saddle point of the coil system as discussed earlier. Guides were made to regulate the direction of vibration and also preventing the sample to stick to one of the poles at high magnetic field.

The measurement conditions were:

- Frequency of input signal= 134 Hz.
- Amplitude of input signal= 5 volts (p-p).
- Time constant = 3 ×100 ms
- Time delay = 3 seconds
- Sensitivity 5 × 10 mV

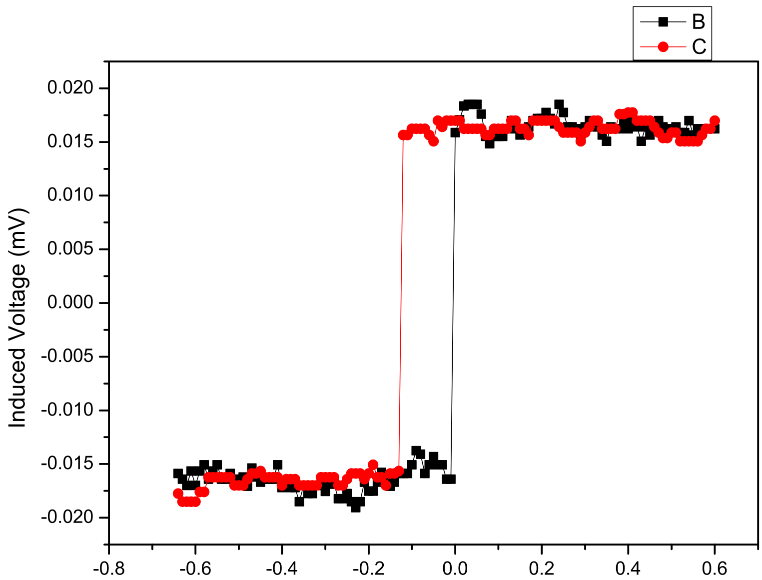


Figure 4.7: Hysteresis loop fo Nickel

Sucess

- Induced signal was good
- Signal was independent of sample geometry and position considerably.

Failures

- Hystereis loop obtained was not close. Many values at lower magnetic fields were missing.
- Sample mounting was very tedious, and time consuming.
- No space to fit in new temperature set ups.

4.2.3.3 Coil design 4.

The advantages with this coil were many. Before going to enumerate the results, a hysteresis loop for nickel using this coil is shown, with the derivation of calibartion factor being followed.

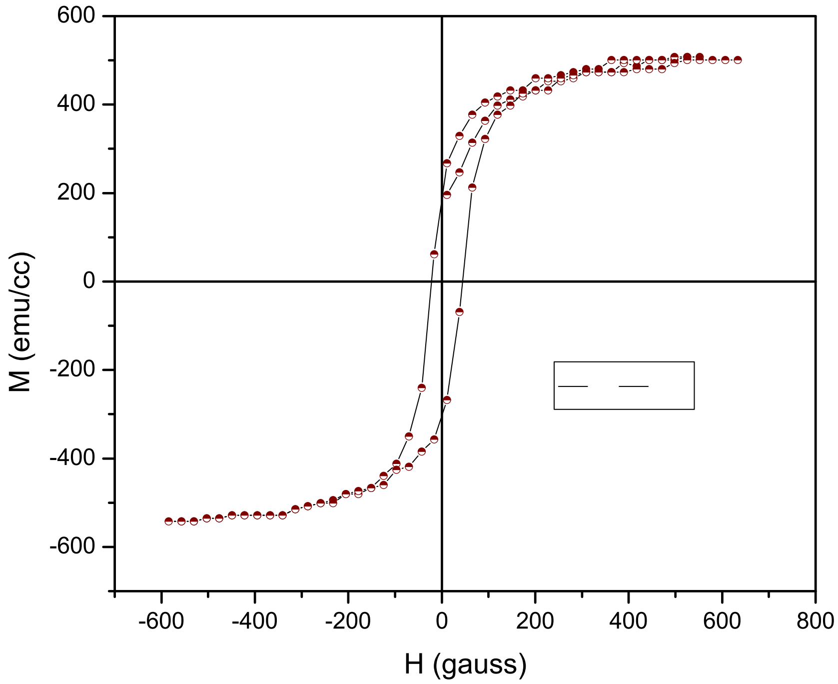


Figure 4.8: Hysteresis loop for Nickel using coil # 4

From the graph above, it can be observed that the signal induced is good and relatively high. The loop obtained is also closed and free from any noisy signals. The saturation of the sample can also be easily observed.

So, before going on other measurements, the instrument was needed to be callibrated using this data.

4.2.3.4 Calibration of the VSM.

The induced voltage in the detection coil not only depends on the relative displacement of the coil and sample, but also on other factors like the dimension of the coil, geometry of the coil, size of the sample, position of the sample demagnetizing factor of the sample, the resistance of the detection coils, the position of the detection coils.[10] . To numerically calculate the induced voltage in the detection coil is not at all possible for sensitive measurements.

The final calibration of the set-up is done using standard samples with known magnetic properties. During calibration the following precusrors were taken care of:

1. Induced signal is not affected by small changes in sample position and deviation from ideal spherical symmetry of the sample.

2. During the run time of the experiment, the frequency and amplitude of input signal are unchanged.

3. The signal voltage is a linear function of the sample magnetic moment.

Thus the calibration factor can be found from the room temperature saturation magnetization value of Nickel.[11]

As the density of Nickel is 8.902 gm/cm^{3}the saturation magnetization can be evaluated as:

$$M_s = \left(\frac{0.60 \times \mu_B}{atom}\right)\left(\frac{9.27 \times 10^{-24} A - m^2}{\mu_B}\right)\left(\frac{0.9135 \times 10^{29} g}{m^3}\right)$$

$$= 5.08 \times 10^5 A/m$$

$$= 508 emu/cm^3$$

So, looking back at the induced voltage data,

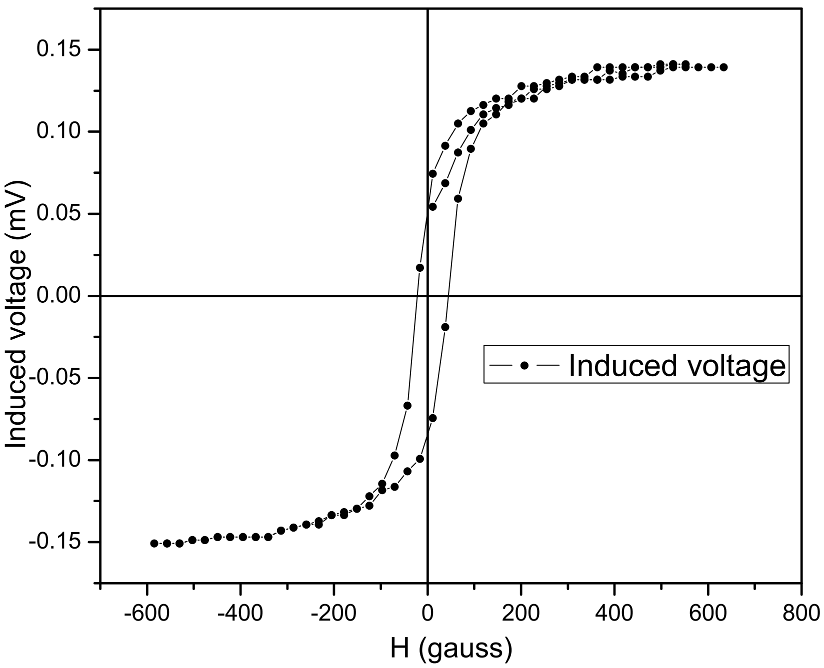


Figure 4.9: Induced voltage loop of Nickel for calibration

Thus the calibration constant was evaluated as:

$$CC = \frac{V_{saturation}}{M_{saturation} \times Volume}$$

so for Nickel sample used, the calibration constant was found out to be:

$$CC = \frac{0.18304406mv}{508emu/cc \times 4.175 \times 10^{-3}cm^3}$$

$$= 0.086304898mv/emu$$

Thus the induced signal is divided with the calibration factor to give us the signal in terms of emu and not in mV. The result for the same Nickel data is shown below:

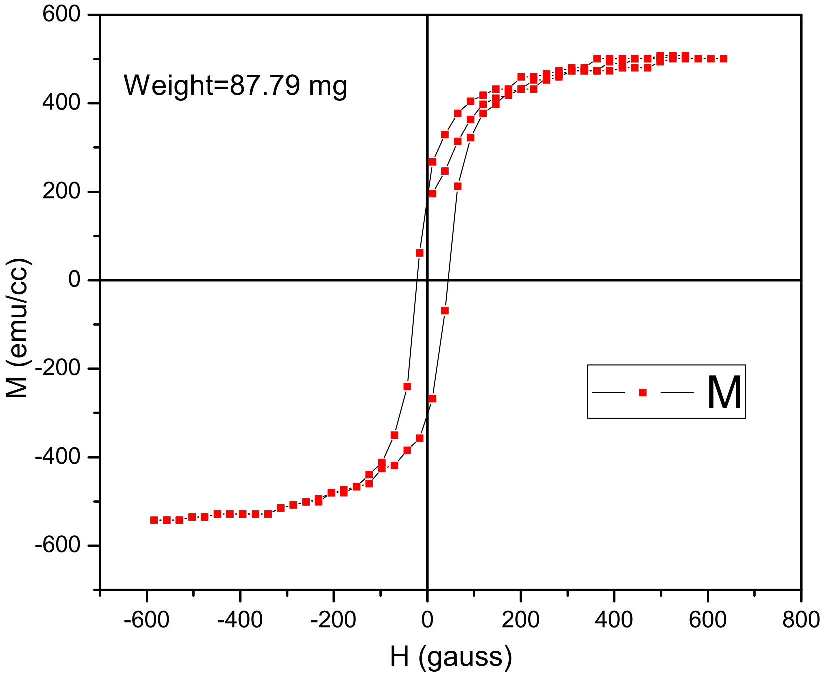


Figure 4.10: Nickel data after calibration

4.2.3.5 Measurements of other samples.

The VSM designed was also used to study the variation of magnetization as a function of temperature for Cobalt Ferrite nano- particles, doped with Platinum.

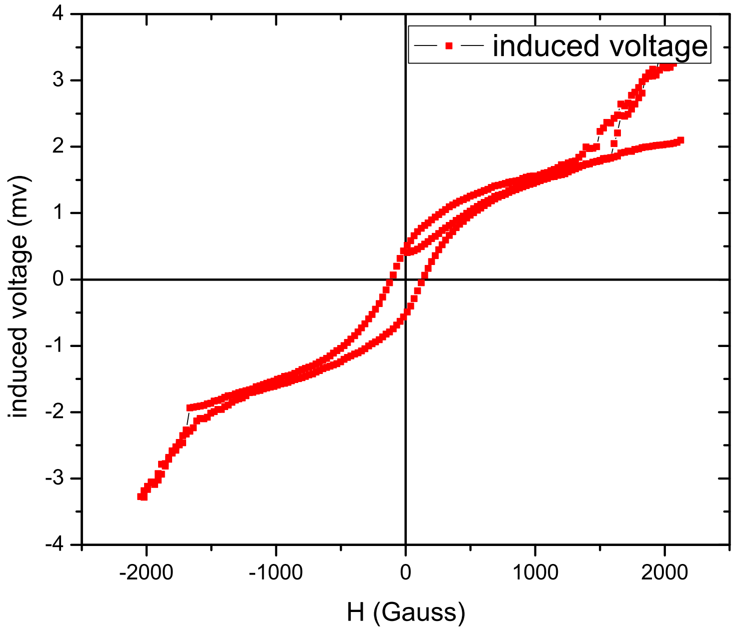


Figure 4.11: Hysteresis loop for Cobalt Ferrite doped with Pt

The hysteresis for soft iron in terms of induced voltage and input current is given below:

4.2.3.6 Advantages of using coil # 4.

After having discussed the results and calculation of calibration factor of the instrument, the advantages and disadvantages of this detection coil can be elisted below:
Advantages

- Since the coils are joined to the poles of the magnet, their position with respect to the sample can be changed. Hence the sensitivity and magnitude of induced voltage can be adjusted as per convenience. For better sensistivity, the coils are pushed close to the sample. Also the entirity of the coil remains with the magnetic field, so sensitivity of the detection coil is preserved.

- Easy to change samples, and mount new samples. To replace samples, only the magnet poles are to be pulled inwards, leaving the rest of the system untouched.

- Enough space for other temperature variation set ups.

- Ratio of sample vibration to the coil area is less, unlike other coils.

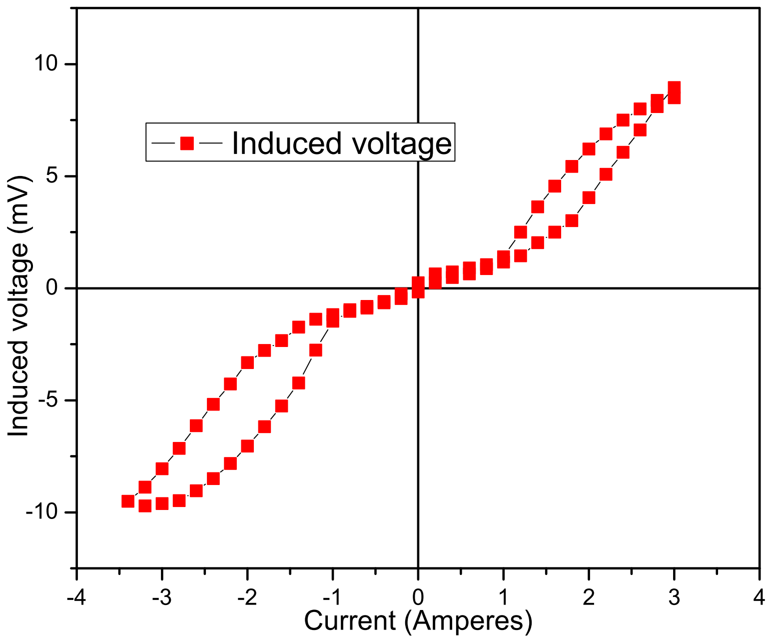


Figure 4.12: Hysteresis loops for Soft Iron

- More relay coils can be incoroprated to make 4 coil or 8 coil arrangement, thus improving sensitivity of induced voltage signal.
- The induced signal is not affected by sample position and sample geometry.

Disadvantages

- Induced signal can be further amplified.
- The coil resistance can be affected at high magnetic fields and low temperatures.
- Extrenal guides required to maintain linearity of sample vibration.

4.2.3.7 Temperature variation measurements.

To acheive measurements of magnetization as a function of temperature, a low- cost temperature variation set- up had been designed which has been discussed earlier in Chapter 3.

The sample and working conditions for measurements were:

- Sample= $La_{0.8}Sr_{0.2}MnO_3$
- Temperature = 273 K
- Time constant= 1 $\times$1 sec
- Sensitivity = 5 $\times$100 μV
- Frequency = 134 Hz.
- Amplitude = 5 v (p-p).
- Time delay = 10 sec.

The sample position is as below:

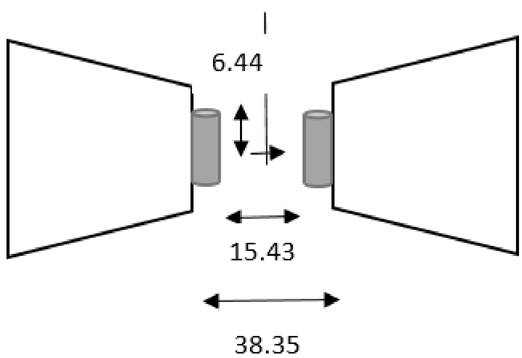


Figure 4.13: Sample position for low temperature measurement

The temperature was monitored by a Pt -100 sensor for which the resistance values were converted to give us measurements in temperature scale.

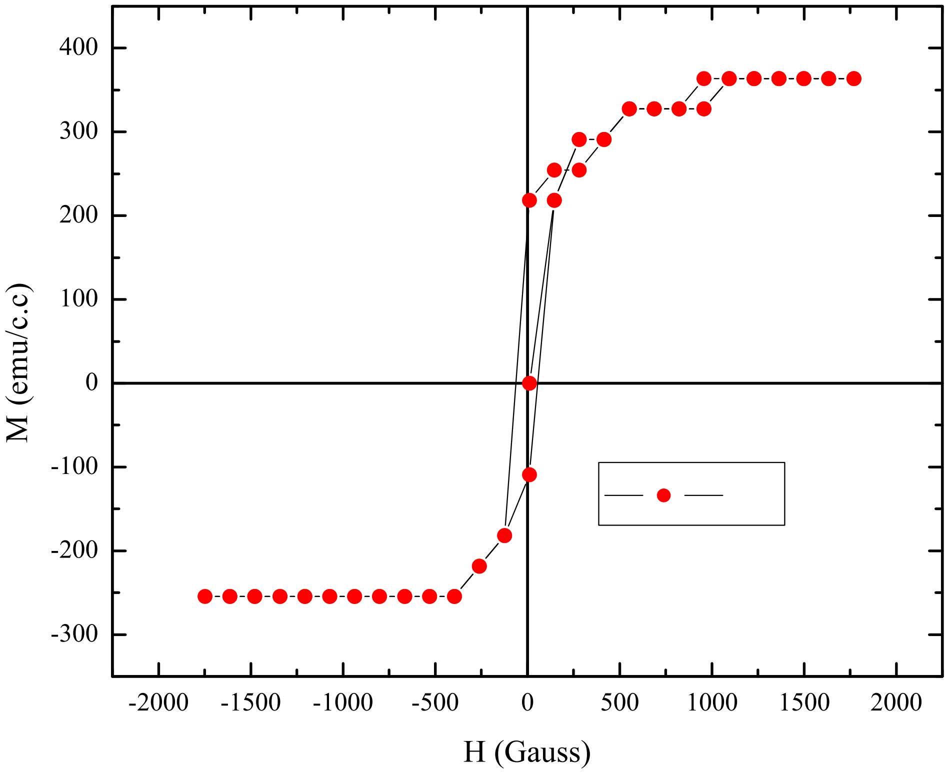


Figure 4.14: Hysteresis loop for $La_{0.8}Sr_{0.2}MnO_3$at 273 K

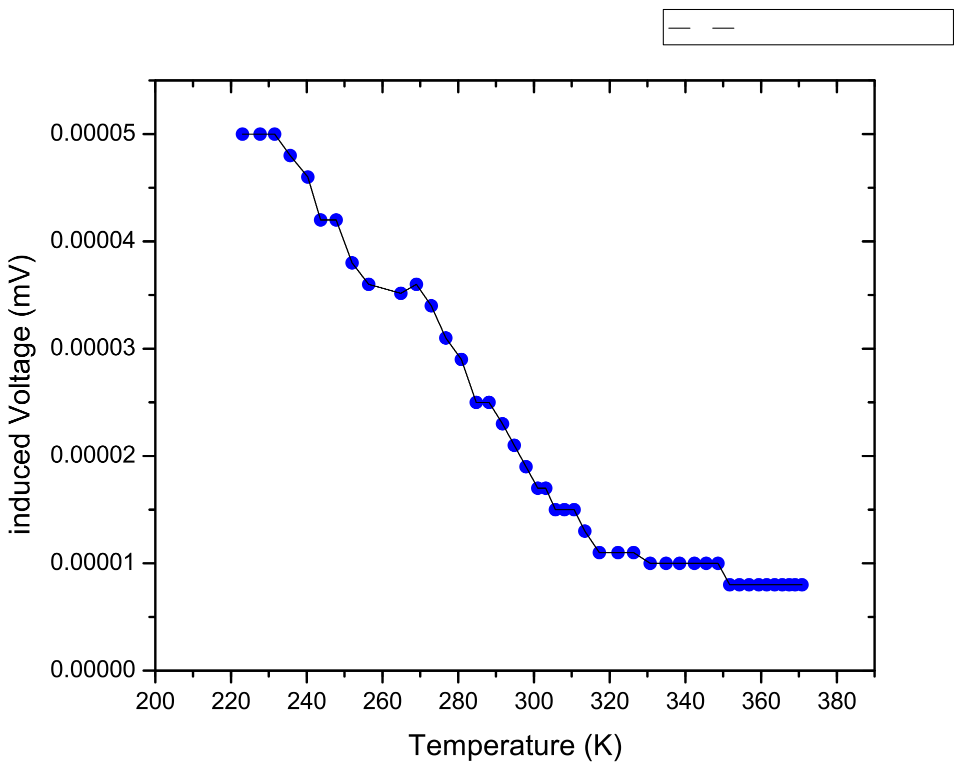


Figure 4.15: Variation of M with temperature for $La_{0.8}Sr_{0.2}MnO_3$

Chapter 5

Conclusion.

5.1 Conclusion

A low cost Vibrating Sample Magnetometer (VSM) is fabricated using a speaker and lock-in amplifier. The VSM is callibrated against a standard Nickel sample. For the testing of the instrument various oxides based samples are measured for their magnetization as a funnction of magnetic field as well as temperature. The obtained results are in good agreement with the reported literature. This implies the sucessful working of fabricated VSM.

The specification and summary of the designed VSM in enumerated below:

- Temperature range: 150 K to 400 K
- Magnetic field: 0.5 Tesla Magnetic field ramp rate determined by magnet and power supply.
- Temperature and magnetic field can be easily ramped.
- Longitudinal configuration= easy to change sample
- Sample mass $\leq$1 g
- Oscillating frequency = 134 Hz .
- Oscillating amplitude range: 0.1 mm to 1mm .
- Accuracy- 0.079 emu.
- Resolution=1.9 $\times 10^{-6}$mV.

5.2 Limitations

1. Operating frequency was 134 Hz. Commercial VSMs operate at frequency less than 50 Hz.
2. Vaccum measurements were not incorporated.
3. Limited amplitude of vibration < 1mm.
4. Magnetic field was upto 2500 Gauss. Could not be used for materials such as Iron, Cobalt , with higher values of saturation magnetization.

5.3 Future scope.

1. A high field strength magnet can be employed for weakly magnetic samples.
2. Vacuum casing can be incorporated for low/ high temperature variation.
3. Mechanical vibrator can be used for low frequency measurement.
4. An amplifier can be designed for amplifying induced voltage.
5. Sample mounts can be developed.

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